MOLECULAR CONTAMINATION IN ENVIRONMENTAL TESTING AT GODDARD SPACE FLIGHT CENTER

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ABSTRACT: A discussion of the various techniques used to detect and analyze materials outgassing from a spacecraft during the environmental test is presented. In addition, a number of compounds that have been detected and their parent outgassing sources are listed. The two major methods for reducing the threat of contamination (i.e., proper choice of spacecraft materials and proper operation of the test facility) are also discussed.

KEY WORDS: contamination, outgassing, polymeric materials, residual gas analysis, vacuum, test facility, chemical analysis, organic residues

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This paper describes the techniques presently being used at Goddard Space Flight Center (GSFC) to detect and minimize molecular contamination of spacecraft or spacecraft subsystems and experiments during the environmental test.

The methods described are an outgrowth of work done to determine the test readiness of oil-diffusion-pumped test chambers for spacecraft having UV reflective optics.

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DEFINITION

Molecular contamination can be defined simply as a deposition of molecules upon a surface that would affect some physical property of that surface (i.e., reflectance, transmittance, absorptance, or surface resistivity). These contaminants are chiefly composed of water vapor and organic molecules of medium to high molecular weight (50 to 800 atomic mass units) and low vapor pressure. The low-molecular-weight organic compounds and residual gases are too volatile to adhere to any surface in a vacuum unless the surface temperature is in the order of -60 C or lower. Hence, these compounds do not ordinarily present a problem since spacecraft surface temperatures are generally above this level.

However, recently there have been some spacecraft experiments flown that utilize detectors that are cooled to 0 C or lower. In this temperature range, some gases, volatile, low-molecular-weight compounds, and water vapor will condense, thus obscuring the detector's view.

EFFECTS OF CONTAMINANTS

Molecular contamination can affect spacecraft performance in four major ways.

1. Loss of Optical Reflectance

A film of silicone oil approximately seven molecular layers (0.4 $\mu g/cm^2$) thick will cause a loss of approximately 8 percent in the reflectance measured at 1216Å (1).[3] Thus, the output of an optical experiment operating in the ultraviolet region in which the beam is reflected from several consecutive reflective surfaces can be seriously reduced.

2. Loss of Optical Transmittance

If the mass accretion upon an optically transmissive surface is sufficient, it can reduce optical and near infrared transmittance (0.4 to 18 μm region). This may result in the degradation of the output of solar-cell arrays, the loss of sensitivity of optical experiments or sensors, or the loss of vision, as in the case of the Gemini 11 where severe clouding of the spacecraft window occurred (2).

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[3]The numbers in parentheses refer to the list of references appended to this paper.

3. Degradation of Thermal-Control Coatings

The molecular contaminants that condense on thermal-control surfaces can alter the solar absorptance-to-emittance ratio. At this time, however, the relationships between the type and amount of molecular contaminant and the extent of degradation for polymeric materials are not clearly understood.

4. Malfunction or Failure of Electronic Systems

Outgassed contaminants condensing on a circuit board assembly, on open contacts, or in waveguide subassemblies can alter effective circuit impedances to the point that malfunction or failure of an experiment or subsystem may result. In addition, molecules outgassing in a confined enclosure may maintain pressure in the corona region. Consequently, when sensitive experiments, transmitters, and high-voltage power supplies are commanded on, they can fail in orbit due to high-voltage corona breakdown if these circuits are turned on before the volatized organics and residual gases have had a chance to diffuse into space.

SOURCES OF MOLECULAR CONTAMINATION

In any specific test there are two basic sources of contaminants: the test facility and the spacecraft.

1. Possible Test Facility Contamination Sources

- Compounds adsorbed on chamber wall from previous tests.
- Cracked oils from the diffusion or mechanical pumping system.
- Paint volatiles or cutting oils used in spacecraft mounting fixtures.
- Outgassed compounds from cabling, potting compounds, adhesives, flexible heaters, etc., used to support the test.
- Water or other compounds adsorbed from the ambient air environment.
- Cleaning solvents.

2. Possible Spacecraft Contamination Sources

- Outgassed compounds from cabling, potting compounds, sealants, circuit boards, adhesives, etc., in the electronic subsystems.

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- Paint volatiles from improperly cured thermal coatings.
- Water or other compounds adsorbed from the ambient air environment.
- Cleaning solvents.

METHODS OF MEASUREMENT

The following complementary analytical techniques are available for the quantitative and qualitative analysis of contaminants outgassed during environmental test:

- Residual gas analysis (RGA).
- Infrared spectroscopy (IR).
- Gas chromatography (GC).
- Gas chromatography-mass spectrometry (GC-MS).
- Quartz-crystal microbalance gravimetry (QCMB).
- Ultraviolet reflectance.

The use of all of these techniques may be necessary for the environmental test of a large spacecraft.

1. Residual Gas Analysis

A residual gas analyzer (Fig. 1). is used to quantitatively and qualitatively identify the molecules present in the spacecraft test environment. This analyzer is a mass spectrometer of relatively low resolution and mass range that ionizes molecules present in the environment and separates them by atomic mass. The data presented by this instrument is in the form of a plot of the molecular weight of the molecules detected versus their relative abundance or partial pressure. A typical RGA spectrum is shown in Fig. 2.

A cursory inspection of the spectrum is all that is required to determine the presence and amounts of residual gases such as air, nitrogen, and water because of their relatively simple molecular formulas.

However, to determine the presence of the higher molecular weight contaminants, those molecules that originate from the spacecraft materials, one must have prior knowledge of the characteristic mass spectra or "fingerprint" spectra of all compounds likely to be found in the test environment. The problem is further complicated by the fact that the mass spectrum presentation represents a summation of the mass spectra of each individual compound in the test environment at that particular instant. The resolution of this mass spectrum

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into its individual compounds requires a sophisticated computer program. Such a program has recently been developed for this purpose (3).

Fingerprint mass spectra of known compounds are fitted to the unknown mass spectrum. The computer then determines the combination of fingerprint spectra that gives the best fit to the unknown spectrum and prints out the results. The use of this program thus far has been limited only by the sensitivity of our analyzers and the limited amount of available fingerprint spectra of commercial compounds.

2. Infrared Spectroscopy

A cold plate (Fig. 3) located in the test volume is operated at 77 K for the duration of the test, during which it collects a fraction of all condensible contaminants in the test environment. At the conclusion of the test, the cold plate is washed with isopropanol solvent.

The mixture of solvent and contaminant is then transferred to the analytical chemical laboratory where the solvent is evaporated and the residue (i.e., contaminants) is analyzed, chiefly by infrared spectroscopy. In this technique of analysis, the infrared spectra of the contaminant is taken in the range from 2.5 to 25 μm. Figure 4 shows a typical infrared spectrum obtained from the OAO thermal-vacuum test. The absorption pattern in this band is representative of the chemical bonds between atoms of these molecules and is thus a fingerprint of the molecules.

However, as with RGA, the resulting infrared absorption spectrum represents a summation of all outgassed compounds that have condensed on the cold plate. Thus, the mixture spectrum is capable of giving general information on the functional groups present: aliphatic, aromatic, hydrocarbon, silicone, ester, amide, etc. For a more complete identification of the individual compounds in the mixture, the mixture must either be separated for individual identification or a computer solution, similar to the one used for mass spectral identifications, must be applied to the mixture spectrum.

3. Gas Chromatography

The gas chromatograph is an analytical instrument that separates a mixture of compounds into pure compounds. The sample obtained from the residue from the cold plate is vaporized and bled into a stream of helium that then passes through a column of absorbing material. The column packing absorbs the less volatile compounds more readily than it absorbs the more volatile compounds. Thus, the material will emerge from the column as a series of time-spaced peaks

in the helium stream with each peak representing a unique compound.
These peaks are usually detected by either flame ionization or thermal
conductivity detectors at the column exit or both.

The results are displayed as a series of roughly gaussian peaks
plotted versus time on a strip-chart recorder. The data are taken as
time of peak height (column traverse time) versus area under the
curve, which is proportional to the amount of the compound. Though
this technique does give a very good fingerprint, it, unfortunately,
does not uniquely determine a given compound and may and usually
does vary somewhat from run to run. Hence, the gas chromatograph
cannot be used for identification without running reference compounds
or trapping the eluted compound for subsequent infrared or mass
spectrometric analysis.

4. Gas Chromatography-Mass Spectrometry

In addition to the infrared spectrometer and gas chromatograph,
the analytical laboratory utilizes a combination gas chromatograph-
mass spectrometer for analysis of the contaminant residue. This
combination overcomes the difficulties of the use of the gas chroma-
tograph alone. The combination enables one to separate the mixture
into its pure molecular constituents and to obtain a mass spectrum
for each constituent from 10 to 1000 atomic mass units.

As a consequence of this capability, thirty or more mass spectra
may be generated from an average cold-plate sample containing a
mixture of up to thirty compounds. For expediting the resolution of
these spectra, automatic data reduction is a necessity. Figure 5
shows the gas chromatograph-mass spectrometer data acquisition
unit in use at GSFC.

5. Quartz-Crystal Microbalance Gravimetry

The QCMB measures gravimetrically the mass accretion of con-
taminant over the surface of an oscillating crystal. The change in the
frequency of oscillation of the crystal is directly related to the mass
accreted upon it. With proper precalibration and temperature com-
pensation, this instrument[4] is capable of measuring as little as
01 $\mu g/cm^2$.

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[4]Hanyok, J., "Crystal Microbalance Vacuum Contamination Monitor,"
NASA X-Document X-324-69-404, National Aeronautics and Space
Administration, Goddard Space Flight Center, Greenbelt, Md.
Sept. 1969.

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If the QCMB is placed at or near a critical surface, such as an optical or thermal-control surface, and the QCMB crystal is configured so as to have the same "view" relationship and temperature as the surface in question, then the mass accretion on the QCMB will approximate the mass accretion on that surface. Figure 6 shows the QCMB system.

6.   Ultraviolet Reflectance

As mentioned previously, small amounts of condensed contaminants will cause a significant loss of reflectance in the ultraviolet region. Hence, with proper precalibration, a mirror sample (Fig. 7) can be used to measure directly contaminant levels on a critical surface. As in the case of the QCMB, the mirror must be configured so that it has the same "view" and temperature relationship of the surface in question. Pretest and post-test reflectivity measurements are made at 1216Å with a vacuum monochromator. Most organic chemicals absorb ultraviolet radiation at this wavelength but in varying amounts, depending on their chemical structure. Consequently, some prior knowledge of the type of contaminant is required for accurate measurement.

A change of reflectivity at 1216Å is indicative of contaminant. The exact level of contaminant deposited is, however, somewhat in doubt.

THE RELATIONSHIP BETWEEN CONTAMINANT MEASUREMENTS
AND ACTUAL SPACECRAFT CONTAMINATION

Although each of the measurement methods previously described will give some quantitative picture of the contaminant levels within the test volume, the actual quantities of material outgassed or desorbed from the spacecraft can only be inferred. This is due to the fact that, in the average test, the majority of the outgassed compounds are pumped out and only a small percentage will return to the spacecraft and condense as a surface contaminant. (Figure 8 shows schematically the relationship among these measurement methods.)

As shown by R. Chaun,[5] the fraction of outgassed material actually condensed on the spacecraft is a function of the chamber wall capture coefficient, the spacecraft surface capture coefficient, and the ratio of internal chamber area to total spacecraft surface area.

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[5]Notes from "Vacuum Technology," a course at the University of California at Los Angeles (Extension), Feb. 14-18, 1966.

For a precise measurement of the amount of contaminant condensed on a spacecraft surface (aside from physically wiping the surface which has been done in a few cases, i.e., OAO 2), it would be necessary to precalibrate the particular spacecraft-chamber system. Modeling techniques can be used to determine the relationship of the material condensed on the spacecraft to the quantitative amounts measured by the techniques described previously.

In solar-vacuum testing, the chamber shrouds are at 77 K or lower; therefore, a molecule that desorbs from the outgassing source will be quickly cryopumped on the cold shroud. Consequently, the analysis of the cold finger sample may show only a small percentage of the outgassed compound, and the RGA or reflective mirror sample may indicate no trace of contaminant molecules, depending on their respective "views" and capture coefficients relative to the outgassing source. In fact it has been found at GSFC that the cold finger will often pick up traces of compounds not detected by the RGA. This is due, possibly, to the integrating effect of the cold plate as well as the limited mass range of the RGA.

In the thermal vacuum test, however, the outgassing molecule will collide several times with the warm wall or spacecraft before coming to rest, thus establishing a relatively uniform molecular flux throughout the test environment. Consequently, all contaminant sensors will "view" the outgassing compound(s). However these compound(s) may or may not condense, depending on their relative surface capture coefficients.

CLASSES OF COMPOUNDS OBSERVED

A qualitative identification of the major compounds observed in the test environment is shown in Table 2. These compounds, collected chiefly from the cold plate, are grouped into three classes according to their percentage of the total condensed sample.

In addition to the compounds listed in Table 2, traces of many more compounds associated with plastics have been observed. Compounds such as 2,6-di-t-butyl-p-cresol, biphenyl, and butyl ethyl ether of ethylene glycol, have been observed in quantity but infrequently, and so far, it has not been possible to determine their source.

Backstreaming silicones from the chamber pumping system are conspicuous by their absence. A positive gas chromatographic technique has been developed that is able to detect 1μg or less of tetramethyltetraphenyl-trisiloxane compound (DC 704). With this technique, backstreaming silicones have been observed in only six tests, with over six hundred samples thus far analyzed.

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In general, outgassing levels of polymeric compounds, will be seriously affected by the following factors: their "cure" cycle (i.e., the correct time-temperature relationship following their preparation), the amounts of plasticizer and antioxidents added to the parent compound to modify its basic physical properties to fit the application, and the operating temperature of the compound.

METHODS OF REDUCING CONTAMINATION

1. Choice of Materials

The ideal method for reducing contaminant levels is to reduce the outgassing rate at the source. This can best be accomplished by the proper choice of materials in the design phase.

The criterion that is used at GSFC for nonmetallic spacecraft materials is based on the work of Muraca and Whittick (4) at the Stanford Research Institute for the Jet Propulsion Laboratory. These criteria limit the use of materials to those with less than 1-percent total weight loss (TWL) and 0.1-percent vacuum condensible material (VCM). The VCM value tends to be far more important than the TWL value because the difference between VCM and TWL is accounted for by the light gases and water. Normally, if a material has a high TWL and a low VCM, it still can be used on a spacecraft if the material is free to outgas into space.

At present, the Material R&D Branch at GSFC is continuing the certification of additional nonmetallic materials for inhouse application. This is being done with a highly automated material outgassing facility developed at GSFC and based on the original design of Muraca and Whittick.[6]

Where a specific requirement can only be met by a material with a high outgassing rate, it is recommended that the material or subassembly be preprocessed (i.e., exposed to a high-temperature and vacuum environment before integration into the spacecraft). Thus, the material whose initial gas load is composed largely of gases, water, and lower-molecular weight fractions is "cleaned out." At the present state of the art, it is not possible to prepare a spacecraft free of all sources of molecular contaminants, but the contaminant hazard can be significantly reduced by the use of the correct nonmetallic materials.

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[6]Fisher, A. and Marmelstein, B., "Micro-Volatile Condensable Material System for Polymer Outgassing Studies," NASA X-Document X-735-69-47, National Aeronautics and Space Administration, Goddard Space Flight Center, Greenbelt, Md., Oct. 1969.

2. Operation of The Vacuum Chamber

Several practical steps may be taken to minimize the contamination threat to a spacecraft during environmental test:

- Pretest bake-out. This will remove contaminants condensed on the chamber wall from previous tests. This can best be accomplished by a high-temperature long-term bake-out (100 C for 48 h is a recommended minimum level).

- Maximum pump-speed to test-volume ratio. Cryopanels and high-capacity pumping system will maximize chamber capture coefficients, thus effecting the rapid removal of the contaminant from the test volume.

- Minimum spacecraft-chamber temperature differential. The temperature differential between the chamber wall and the spacecraft during cold-to-hot temperature transition should be held to a minimum, preferably 10 C or less. This will extend transition time; however, it will decrease chamber-to-spacecraft contamination.

- Backfill chamber. In order to hold contaminants that have been trapped on the cryopanel, it is desirable to backfill the test volume to approximately 0.1 torr with dry GN_2 when concluding a test in which large cryopanels are used. This will reduce the molecular mean free path in the test volume to less than 2.5 mm while still maintaining the environment below the region of convective heat transfer. Thus, the probability that a trapped contaminant released by the cryopanel during the warm-up phase will reach the spacecraft is considerably reduced.

- Protective cryopanels A relatively small cryopanel interposed between a sensitive experiment and the chamber cryopanel will occlude the experiment's "view" of the larger chamber cryopanel, thus reducing the contaminant hazard due to outgassing from the chamber wall. In addition, larger cryopanel surfaces that are kept cold during chamber warmup will act as a molecular sink for molecules released from the chamber wall.

- Sweep purging Backstreaming of a diffusion pumping system is at a maximum when the diffusion pump is operating in a transition mode (i.e., pumpdown or repressurization). A small leak or purge of nitrogen bled into the chamber during this transition period will stabilize molecular flow outward (i.e., from the test volume to the atmosphere) (5), thus minimizing the risk of backstreaming.

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CASE HISTORY—AN EXPLORER SPACECRAFT

A contaminant case history is given in the succeeding paragraphs, for one of the Explorer series spacecraft.

1. Spacecraft Description

The spacecraft had a mass of approximately 140 kg and carried six experiments. The nonmetallic materials used in the fabrication of the spacecraft were

* Polyurethane foam potting compounds.
* Epoxy and silicone potting compounds.
* Silicone and ester lubricants.
* Silicone adhesives.
* Epoxy adhesives.

2. Test Procedure

The spacecraft was subjected to ten-day thermal-vacuum test followed by a six-day solar-vacuum test. A three-day thermal-vacuum retest was performed finally to assure that malfunctions that had occurred during the first test had been corrected.

3. Test Instrumentation

* Residual gas analyzers
   Consolidated Electrodynamics Corp. 21-613
   General Electric Monopole
* Cold plate sample analysis
   Perkin Elmer 621 Spectrophotometer
   Hewlett Packard 5750 Gas Chromatograph
   Perkin Elmer 270 Gas Chromatograph - Mass Spectrometer

4. Analysis of Outgassed Compounds

A complex mixture of almost 2.5 g of outgassing products was found on the condenser plate following the first thermal-vacuum test. This sample was analyzed by infrared, gas chromatographic, and mass spectrometric techniques. Figure 9 shows an RGA spectrum taken during the second retest phase, Fig. 10 shows the IR spectrum of the cold plate sample mixture, and Fig. 11 shows the gas chromatogram of the cold plate sample mixture.

The mixture of at least fifty components, twenty of them major in quantity, was found to have the following composition: a complex mix-

ture of aliphatic or alicylic hydrocarbons or both, 50 to 60 percent; a series of phenylmethyl siloxanes, 5 to 10 percent; six members of a homologous series of dimethyl silicones, 15 to 20 percent; and pure bis(2-ethyl hexyl) phthalate, 20 percent. The hydrocarbons could come from several sources such as lubricating oil (i.e., a petroleum-based lubricant) or perhaps from the outgassing of some O-rings, which quite often use plasticizer with white oils of this type. The dimethyl-silicones and phenylmethyl silicones are most likely due to outgassing of RTV and other silicone resins. The bis (2-ethyl hexyl) phthalate is one of the most common plasticizers used in the United States and thus could come from outgassing of various compounds. It should be noted that the use of such compounds is discouraged at GSFC.

This mixture did not contain any of the oil from either the chamber diffusion pumps (Dow Corning 704) or from the mechanical pumps (Sunvis 931). These results were obtained by running Dow Corning 704 and Sunvis 931 through a gas chromatograph under the same conditions as those of the outgassing mixture. The fingerprint obtained did not match any peaks in the outgassing mixture. During these thermal vacuum tests, a sun gun (quartz-iodine photoflood lamp) was used to excite the spacecraft solar-aspect sensor. This sun gun was suspected of being a source of the hydrocarbon contaminants seen in the analysis because of its high operating temperature. Consequently, a separate outgassing test was performed on the sun gun. Gas chromatographic and infrared spectra of the condensed cold plate residue failed to show any similarity with spectra from the thermal-vacuum test. Hence, the sun gun was ruled out as a possible contributor of hydrocarbons.

For the subsequent solar-vacuum test and thermal-vacuum re-test, the condensor plate samples taken after these tests showed almost the same infrared patterns and gas-chromatographic patterns as had been obtained from the thermal-vacuum test, only in much less quantity. Results of the RGA scans were inconclusive during both the initial thermal-vacuum and initial solar-vacuum tests because of a loss of resolution during the thermal-vacuum test and a loss of sensitivity in the solar-vacuum test. In the solar-vacuum test, no peak above atomic mass 57 could be detected.

SUMMARY AND CONCLUSIONS

With the measurement techniques described, it has been possible to partially identify and roughly approximate the contaminants outgassed in a specific test. However, a great deal more analytical and experimental work must be done before exact quantitative and qualita-

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tive contaminant data can be determined. Two general observations may be made:

(1) It has been found that, in most of the tests, the test item is the prime source of contaminants.

(2) Contamination traceable to the chamber diffusion-pump system occurred in a very small percentage of the tests (0.1 percent).

Future spacecraft missions involving extended orbital life such as the Grand Tour deep-space probe and the next generation of communication satellites and orbiting observatories will require a functioning lifetime of three years and longer. This imposes a severe constraint on the spacecraft designer in his choice of materials.

Longer orbital lifetimes also will mean an extended environmental-testing program; consequently, it will be necessary that the environmental test engineer be assured that his test facility is clean and that his methods of operation minimize the contaminant hazard.

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Table 1—Comparison of measurement techniques.

Measurement	RGA	Cold Plate or Wipe Sample (Residue)			QCMB	Mirror Reflectance
		IR	GC	GC-MS		
Minimum Detectable Quantity	Molecular Quantities	$50 \mu g$[a]	$1 \mu g$	1 to $5 \mu g$	0.01 $\mu g/cm^2$	0.4 $\mu g/cm^2$
Range of Measurement Sensitivity	Lower Organics Only	All Organics Which Will Volatize and Condense on Cold Plate[b]			All Organics Which Will Volatize and Condense[b]	
How Measured	Instantaneous				Integrated Over Test	
Type of Measurement	In Test	Post-test			In test	Post-test
What Is Measured	Outgassing Compounds in Environment	Condensed Contaminants				

[a]This is an upper figure; analysis can be performed, if necessary, on as little as a 10 μg sample.

[b]Generally pure compounds with molecular weights over 1000 will not volatize in a vacuum environment and hence will not outgas.

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Table 2—Major compounds observed in the test environment.

Class I (10 to 60%)		Class II (0-10%)		Class III (Trace)	
Compound	Source	Compound	Source	Compound	Source
Phthalate Esters	Common Plasticizer Used in Processing Polymers; Used as a Test Aerosol in Clean Room Filters	Butyl, Decyl Esters of Adipates and Sebecates	Specific Purpose Plasticizers	Ketones	Solvents, Plasticizers in Vinyl Polymers
Dimethyl Siloxanes	Low-Molecular-Weight Species Desorbed From High-Molecular-Weight Silicones Silicone Fluids	Phenylmethyl Siloxanes	Low-Molecular-Weight Species Desorbed From High-Molecular-Weight Special Purpose Silicones Resistant to Radiation and Temperature Extremes (i.e., Adhesives, Diffusion Pump Oils, O-Ring Seals)	Chlorinated Biphenyl and Other Chlorinated Hydrocarbons	Solvents Used for Cleaning, Plasticizers
Saturated and Unsaturated Hydrocarbons	Paraffinic Lubricants and Residual Cutting Oils Solvents	Halogenated Siloxanes	Special Silicones for Various Specific Uses	Glycidyl Ethers and Other Low-Molecular-Weight Epoxy Compounds	Epoxy Resins
				Amines and Amides	Curing Agents From Epoxies Nylons
				Polyesters	Polyurethanes (i.e., Potting Compounds)
				Fluorinated Hydrocarbons	Solvents Used for Cleaning

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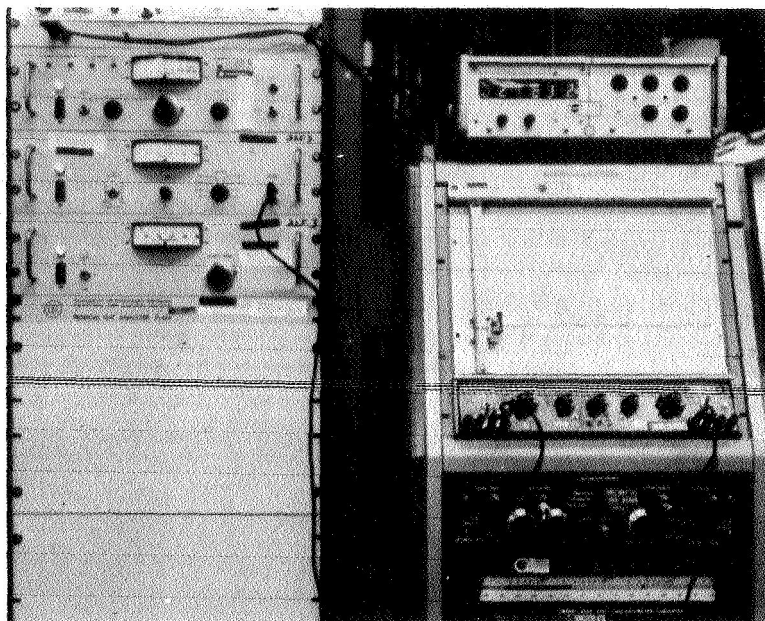
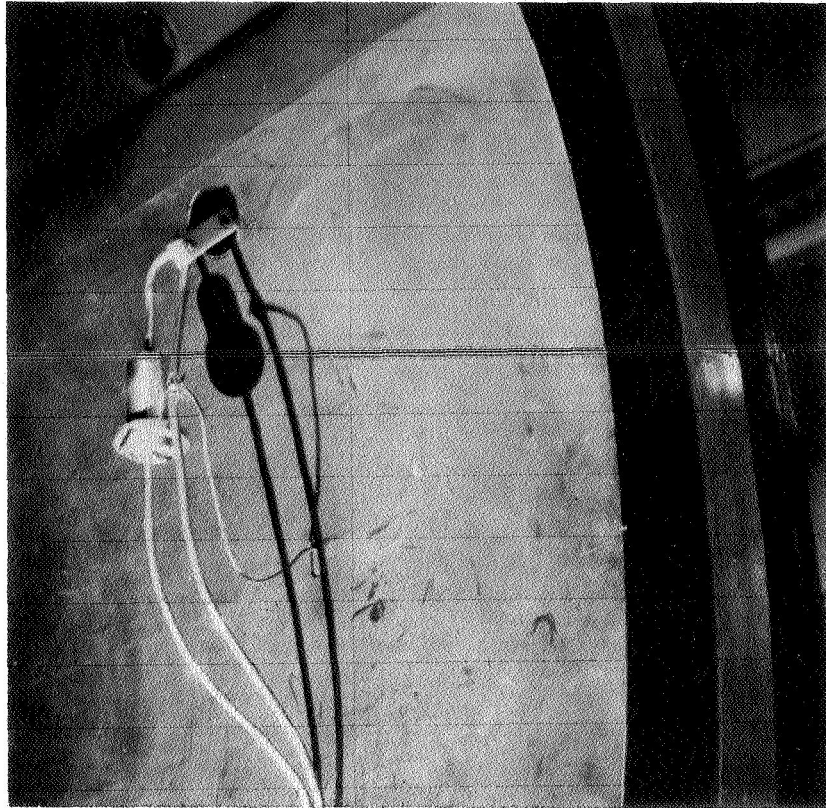Fig. 1—Residual gas analyzer (RGA).
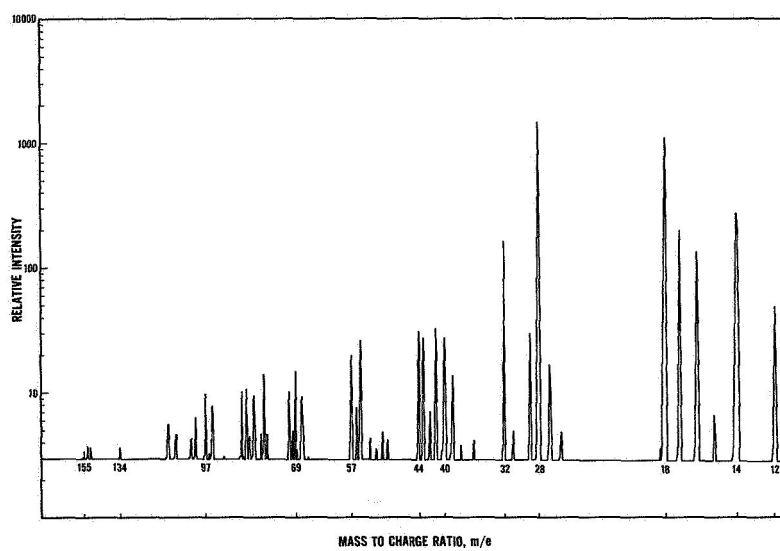
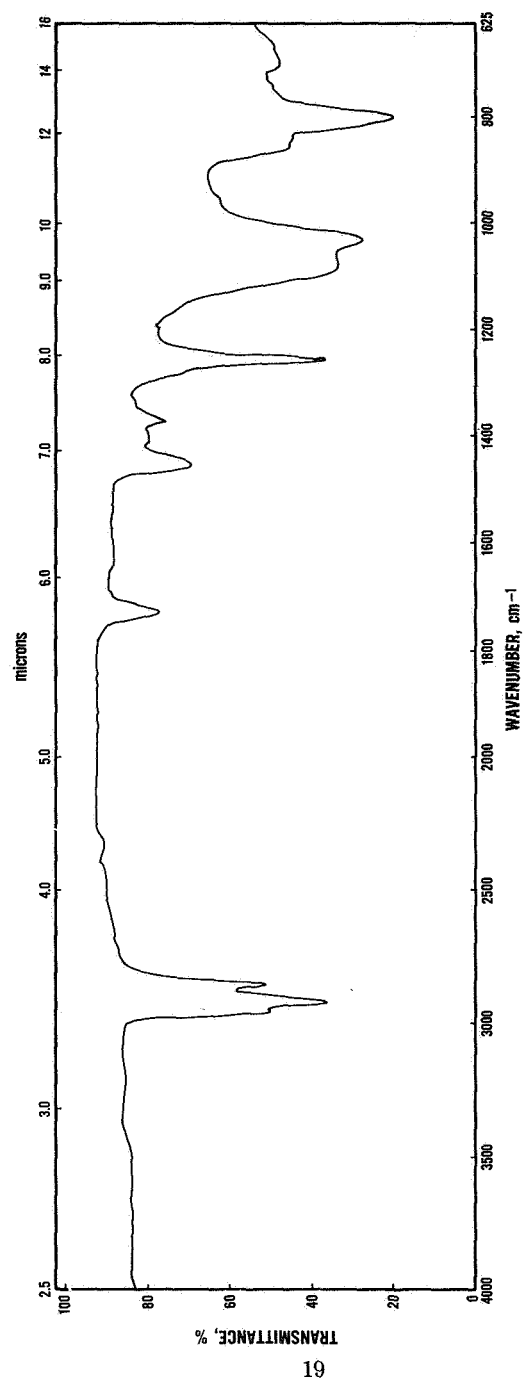Fig. 2—RGA mass spectrum.

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Fig. 3—Chamber cold plate.

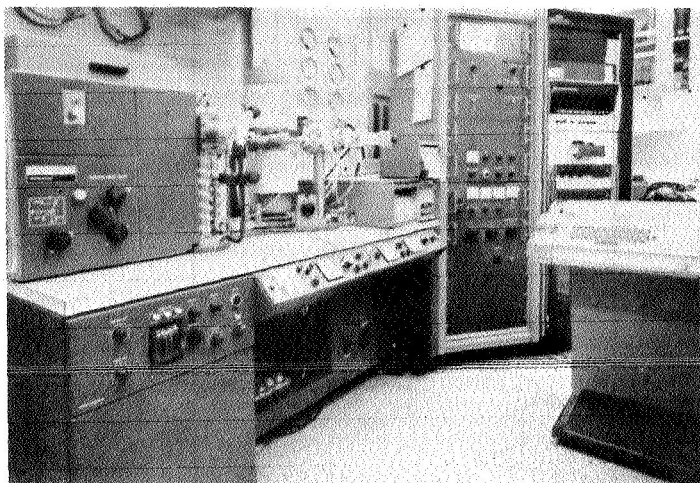Fig. 4—Surface contamination from OAO 2, infrared spectrum.

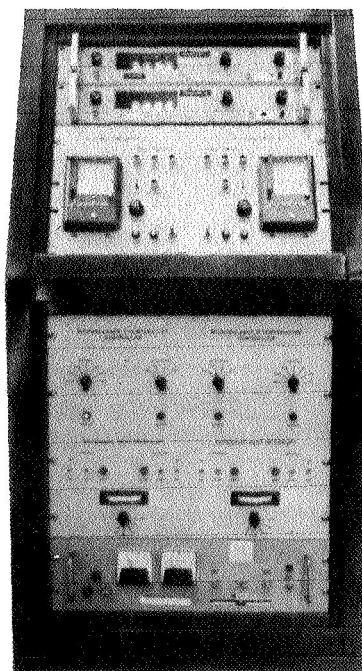Fig. 5—Gas chromatograph-mass spectrometer (GC-MS) and data reduction system.

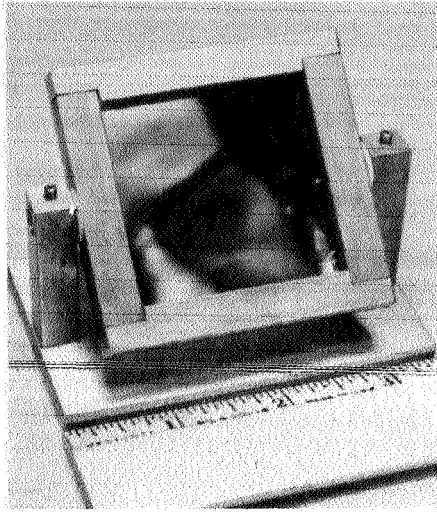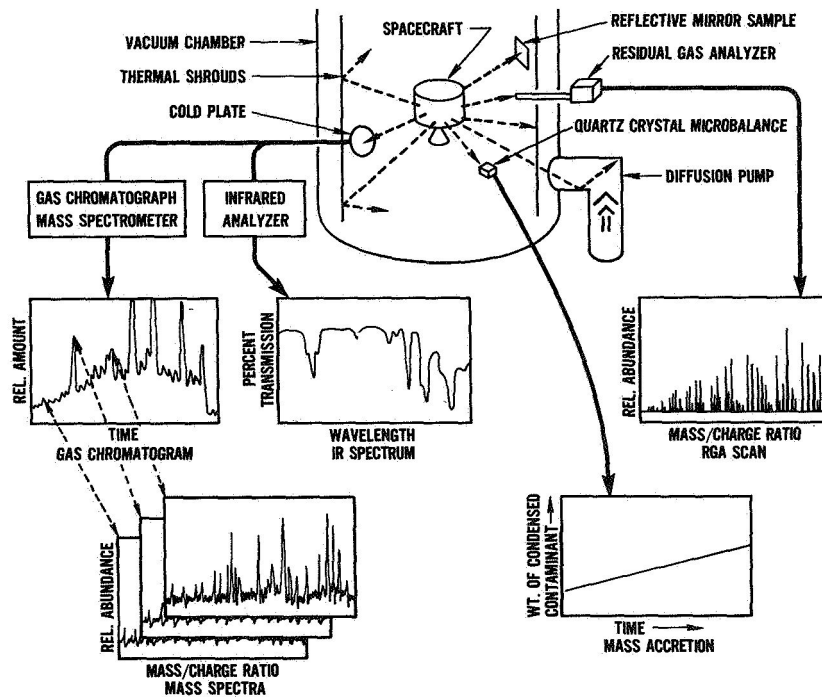Fig. 6—Quartz crystal micro-balance (QCMB) system.

Fig. 7—Mirror sample.

Fig. 8—Relationship of measurement methods.

Fig. 9—RGA spectrum, Explorer spacecraft.
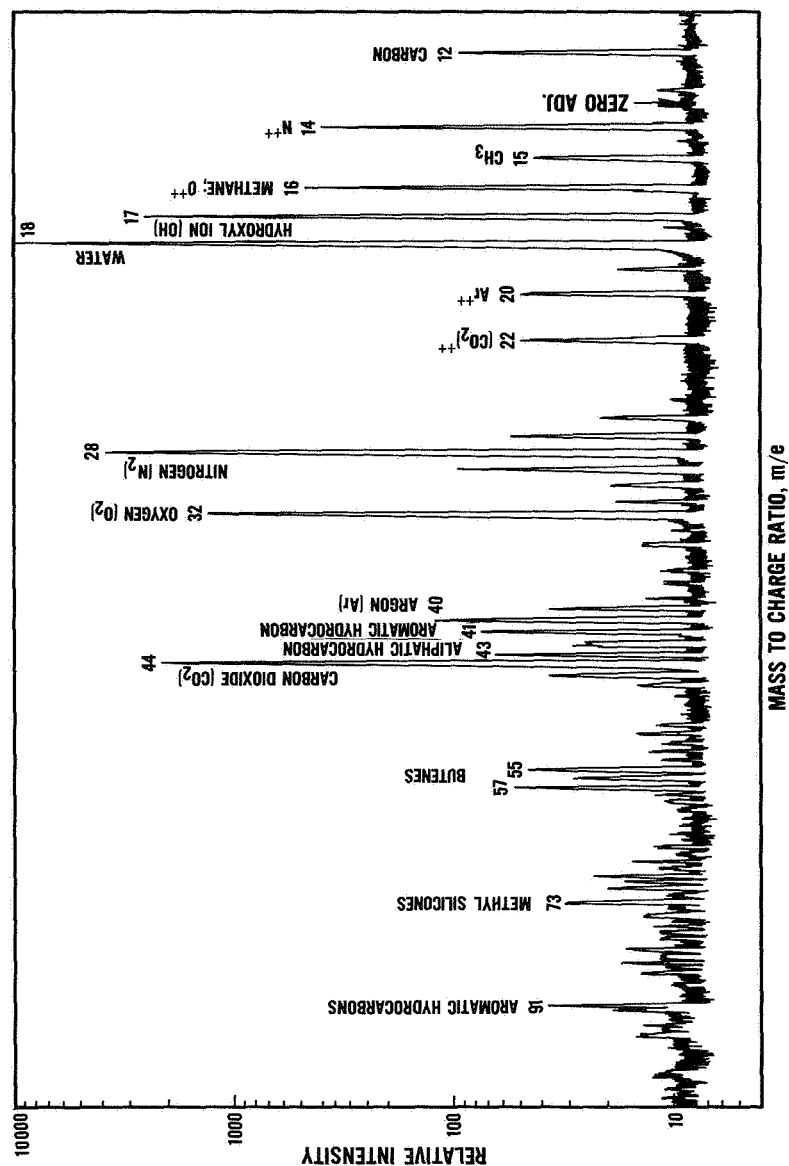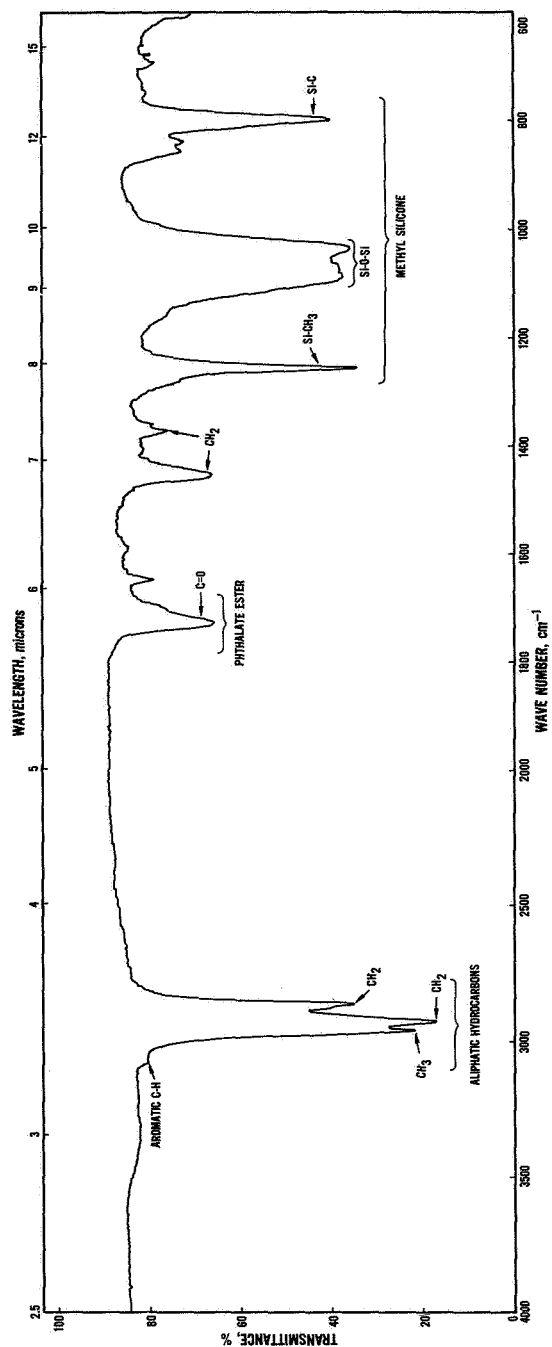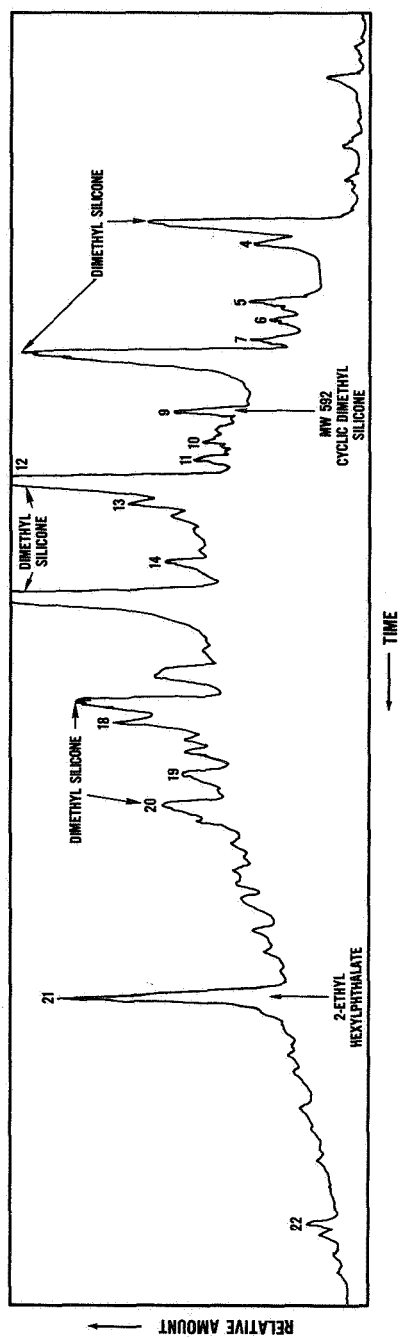
Fig. 10—Explorer cold plate sample, infrared spectrum.

Fig. 11—Gas chromatogram of cold plate sample, Explorer spacecraft.

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